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SALT STRESS CORROSION OF RESIDUALLY STRESSED Ti-8Al-1Mo-1V ALLOY SHEET AFTER EXPOSURE AT ELEVATED TEMPERATURES

by Howard B. Dexter

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AT ELEVATED TEMPERATURES

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Start

SUMMARY

An experimental investigation of the salt-stress-corrosion cracking of residually stressed Ti-8Al-1Mo-1V alloy sheet (duplex annealed) has been carried out with bend specimens which were brake formed from 0.050-inch-thick (1.3 mm) sheet over dies with radii of 0.25, 0.5, 1, 1.5, and 2.5 inches (0.6, 1.3, 2.5, 3.8, and 6.4 cm); residual tensile stresses ranged from 25 to 65 ksi (172 to 448 MN/m^2). Specimens were salt dipped in a 3.4-percent NaCl solution and exposed at temperatures from 400° to 600° F (477° to 589° K) for times up to 6400 hours. Severe stress-corrosion cracking was found to occur after 20 hours exposure at 600° F (589° K) for the 0.25-inch-radius (0.6 cm) specimens, but no salt stress corrosion was noted at 400° F (477° K) regardless of stress. Salt stress corrosion after 3200 and 6400 hours exposure began to occur at temperatures above 400° F (477° K) for the bend radii investigated. Additional tests were run to determine the relative effects of NaCl, $CaCl_2$, $MgCl_2$, sea salt, and simulated sea salt (seven parts NaCl to one part $MgCl_2$) on stress-corrosion cracking. The results showed NaCl to be the most corrosive. Conventional stress-relieving procedures were effective in eliminating stress corrosion when performed in an argon environment, but results were erratic when the specimens were stress relieved in air.

End

INTRODUCTION

Strength, stiffness, and crack-propagation investigations of structural materials for a Mach 3 supersonic transport have indicated that Ti-8Al-1Mo-1V alloy sheet is a potential skin material (refs. 1 and 2). However, the results of references 1 and 3 indicate that this titanium alloy is susceptible to salt stress corrosion at elevated temperatures. Research on this problem at the Langley Research Center has included the effect of various environmental factors (ref. 4) and the effect of residual tensile stresses in brake-formed sheet (ref. 5). Reference 5 has shown that severe salt-stress-corrosion cracking occurs in residually stressed specimens subjected to a 0.125-inch-radius (0.3 cm) bend when the material is exposed at 550° F (561° K) for times as short as 100 hours.

The investigation reported herein is an extension of the work on the salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet (duplex annealed) due to residual stresses (ref. 5). The present paper shows the stress and temperature levels at which stress-corrosion cracking occurs, the effect of various chlorides, and the effectiveness of conventional stress-relief treatments. The results are based upon room-temperature tests of stress-corrosion specimens with bend radii from 0.25 to 2.5 inches (0.6 to 6.4 cm) and residual tensile stresses from 25 to 65 ksi (172 to 448 MN/m^2). These specimens were exposed at temperatures from 400° to 600° F (477° to 589° K) for times up to 6400 hours.

The units used for the physical quantities defined in this paper are given both in the U.S. Customary Units and in the International System of Units (SI) (ref. 6). Factors relating the two systems are given in appendix A.

SPECIMENS AND EXPERIMENTAL PROCEDURES

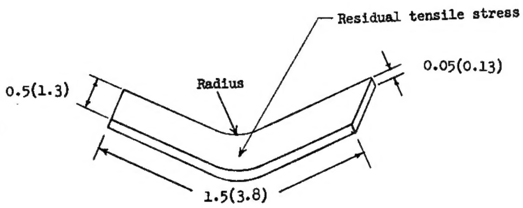
Specimens

The residual stress specimen configuration is shown in figure 1. Specimen blanks were sheared from flat 0.050-inch-thick (1.3 mm) Ti-8Al-1Mo-1V sheet in the duplex annealed condition. Duplex annealing consists of 8 hours at 1450° F (1061° K) with a furnace cool to room temperature followed by 15 minutes at 1450° F (1061° K) with air cooling to room temperature. The flat blanks were run through a commercial vibratory cleaning and deburring process to remove shearing burrs. Inasmuch as this treatment was subsequently found to be effective in preventing stress corrosion, it was necessary to eliminate this vibratory effect in order to study the stress corrosion characteristics. Etching 1/2 mil (0.013 mm) from each surface with a solution of 18 percent HNO_3, 3 percent HF, and 79 percent H_2O, by volume, proved sufficient for this purpose.

The specimens were brake formed at room temperature over dies with radii of 0.25, 0.5, 1, 1.5, and 2.5 inches (0.6, 1.3, 2.5, 3.8, and 6.4 cm) to an initial bend angle of 90° or the maximum angle less than 90° obtainable. The final bend angle varied with the amount of springback, which is a function of the bend radius. The residual tensile stress is a maximum on the inside of the bend of the formed specimens. The table in figure 1 shows the calculated residual tensile stresses for each bend radius. The method used for calculating the residual stresses is described in appendix B.

Salt Coating and Temperature Exposure

Salt coatings were applied by dipping the specimens at room temperature in a 3.4-percent solution of NaCl and distilled water. The 3.4-percent NaCl solution was chosen to represent a typical sea water concentration. After dipping, the specimens were dried in an oven at 200° F (366° K) so that the salt would collect and form a thin film of salt crystals on the surface possessing residual tensile stresses. Salt-dipped and control (no salt) specimens were subjected to



Bend radius		Calculated residual tensile stress	
in.	cm	ksi	MN/m^2
0.25	0.6	65	448
.5	1.3	60	414
1	2.5	55	379
1.5	3.8	50	345
2.5	6.4	25	172

Figure 1.- Residually stressed specimen and calculated residual tensile stresses. Dimensions are shown in inches and parenthetically in centimeters.

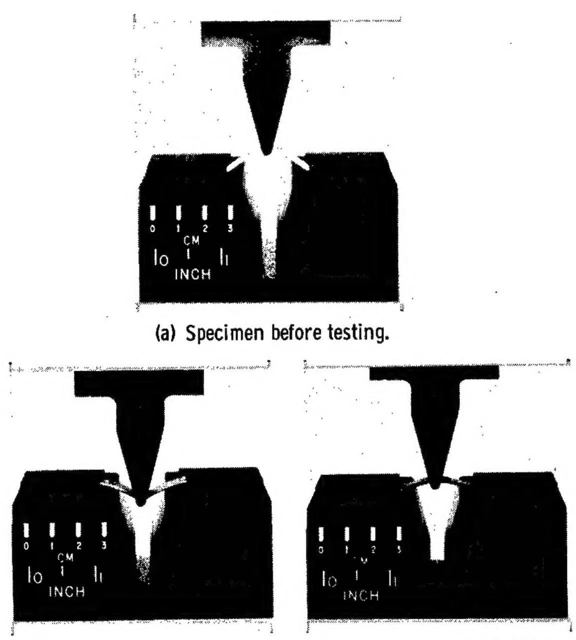
continuous heating at 400°, 450°, 500°, 550°, or 600° F (477°, 505°, 533°, 561°, or 589° K) in circulating air ovens for various lengths of time up to 6400 hours. Specimens were removed from the ovens after selected exposure times and mechanically tested or metallurgically examined.

Other stress-corrosion tests with different types of chlorides or salt were run on some of the 0.25-inch-radius (0.6 cm) specimens exposed at 600° F (589° K) for times less than 1000 hours. Solutions of 3.4-percent pure NaCl, $CaCl_2$, $MgCl_2$, and simulated sea salt (seven parts NaCl to one part $MgCl_2$) were investigated. Some specimens were dipped in sea water obtained from the mouth of the Chesapeake Bay.

Mechanical Tests

The effect of the salt stress corrosion on bend ductility was determined from a reverse-bend test at room temperature. The reverse-bend test setup is illustrated in figure 2. A typical specimen before testing is shown in figure 2(a). A control specimen (no salt), shown in figure 2(b), must be deflected until a reverse curvature is produced in order to cause fracture. The salt-dipped specimens with high stresses (fig. 2(c)) require only a small amount of deflection to cause fracture after exposure at 600° F (589° K). The specimens were tested in a 120-kip (534 kN) machine with load applied at a constant head speed of 0.2 in./min (0.085 mm/s) until fracture occurred. Load-deflection curves were recorded for many of the tests by using an X,Y plotter. Load and deflection at fracture were recorded for each test; head deflection was measured with a dial gage.

The reverse-bend test was selected because of the ease of testing and because it provides a means of determining the presence of stress-corrosion cracks. If cracks develop due to salt, stress, and elevated temperatures, they will initiate



(a) Specimen before testing.

(b) Control specimen near fracture.

(c) Salt-dipped specimen near fracture.

L-65-9037

Figure 2.- Reverse-bend test setup showing specimen during test after elevated-temperature exposure.

on the inside surface of the bend where the residual tensile stress is a maximum. The presence of cracks due to stress corrosion resulted in failure with only a small bend deflection as compared with the amount of deflection required to cause failure when no cracks were present.

Metallurgical Examination

A metallurgical examination to determine the extent and depth of salt-stress-corrosion cracking was made of representative specimens which had been exposed for various times. Specimens were edge mounted in plastic and wet ground to remove 0.125 inch (0.3 cm) of material from the edge of the specimen. The specimens were polished and then chemically etched with a solution of 97 percent H_2O, 2 percent HNO_3, and 1 percent HF, by volume. Crack penetration was measured by using a microscope with a filar micrometer eyepiece.

Alleviation of Residual Stresses

Inasmuch as a tensile stress is required to produce stress-corrosion cracking, the possibility of eliminating stress corrosion in residually stressed Ti-8Al-1Mo-1V alloy sheet specimens by a conventional stress-relieving procedure was investigated. After forming, the 0.25-inch-radius (0.6 cm) specimens were stress relieved at 1450° F (1061° K) for 1 hour in either air or argon, and then cooled in air or argon, respectively. The oxide layer on the specimens stress relieved in air was removed by blasting with fine aluminum oxide grit, followed by pickling in a solution of 18 percent HNO_3, 3 percent HF, and 79 percent H_2O, by volume. The specimens stress relieved in argon were pickled only. Specimens were salt dipped in a 3.4-percent $NaCl$ solution and subsequently exposed for various times at 600° F (589° K). Control specimens were also tested to see whether the stress-relief process had any marked effect on the stability of the material. Several groups of specimens were run to determine whether the results would be consistent.

RESULTS AND DISCUSSION

Characteristics of the Reverse-Bend Test

Typical load-deflection curves recorded at room temperature after exposure at 600° F (589° K) are shown in figure 3. Figure 3(a) shows load-deflection curves for 1-inch-radius (2.5 cm) specimens exposed for times up to 400 hours at 600° F (589° K). The sudden change in slope at approximately 0.2-inch (5 mm) deflection occurs at the point where the specimen is flattened out; an increase in load is required to initiate the reverse curvature. The load-deflection curves are then smooth up to a maximum load where the specimen yields slightly before fracture occurs. When salt-stress-corrosion cracking occurs, the amount of deflection at failure decreases considerably with increasing exposure time.

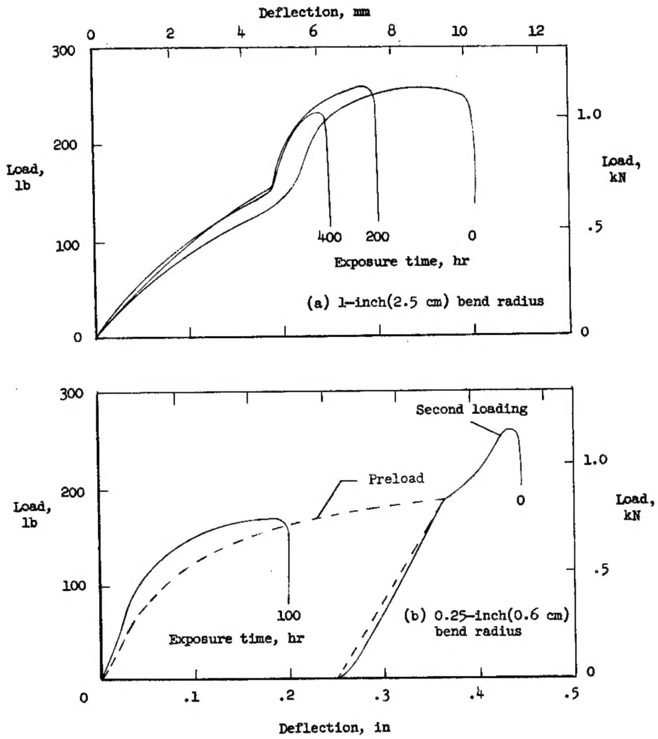


Figure 3.- Load-deflection curves for duplex-annealed Ti-8Al-1Mo-1V residually stressed corrosion specimens at zero exposure and after various exposures at 600° F (589° K).

Figure 3(b) shows typical load-deflection curves for the 0.25-inch-radius (0.6 cm) specimens for zero and 100-hour exposures at 600° F (589° K). The specimens exposed for 100 hours fractured before they completely flattened out; therefore, bending to a reverse curvature was impossible and no sudden change of curve slope was obtained. Neither the control nor the zero-exposure specimens with 0.25-inch (0.6 cm) or 0.5-inch (1.3 cm) radii could be tested according to the method illustrated in figure 2 because of specimen slippage. Consequently, it was necessary first to preload these specimens to a deflection of approximately 0.25 inch (0.6 cm) with a flat ram before testing them in the reverse-bend setup shown in figure 2. The dashed curve in figure 3(b) represents the preloading portion of the test, whereas the solid curve illustrates the secondary loading in which the specimen fractured in reverse bending. Figure 3(b) shows the large difference between the amounts of deflection required to produce fracture for the 100-hour-exposure specimens and the zero-exposure specimens.

Effect of Various Bend Radii and Temperatures

The relative susceptibility of the 0.25-inch-radius to 2.5-inch-radius (0.6 cm to 6.4 cm) specimens to salt stress corrosion at temperatures from 400° to 600° F (477° to 589° K) is shown in figures 4 to 8. The curves shown in these figures are drawn through the approximate lower limit of the test data. The increasing severity of salt-stress-corrosion cracking with exposure time is shown as a reduction in the relative deflection. Relative deflection is defined as the ratio of the deflection at maximum load for exposed specimens to the deflection at maximum load for unexposed specimens.

The 0.25-inch-radius (0.6 cm) salt-dipped bend specimens exposed at 600° F (589° K) (fig. 4(a)) are the most severely damaged specimens, as would be expected. The results indicate that salt-stress-corrosion cracking may be expected after only 20 hours exposure at 600° F (589° K) with the 0.25-inch-radius (0.6 cm) specimens (calculated residual tensile stress of 65 ksi (448 MN/m^2)). In general, the severity decreases with an increase of bend radius (decrease of residual stress) and a decrease in temperature. The figures show that no detectable salt stress corrosion occurs at 400° F (477° K) regardless of the magnitude of bend radius or the exposure time.

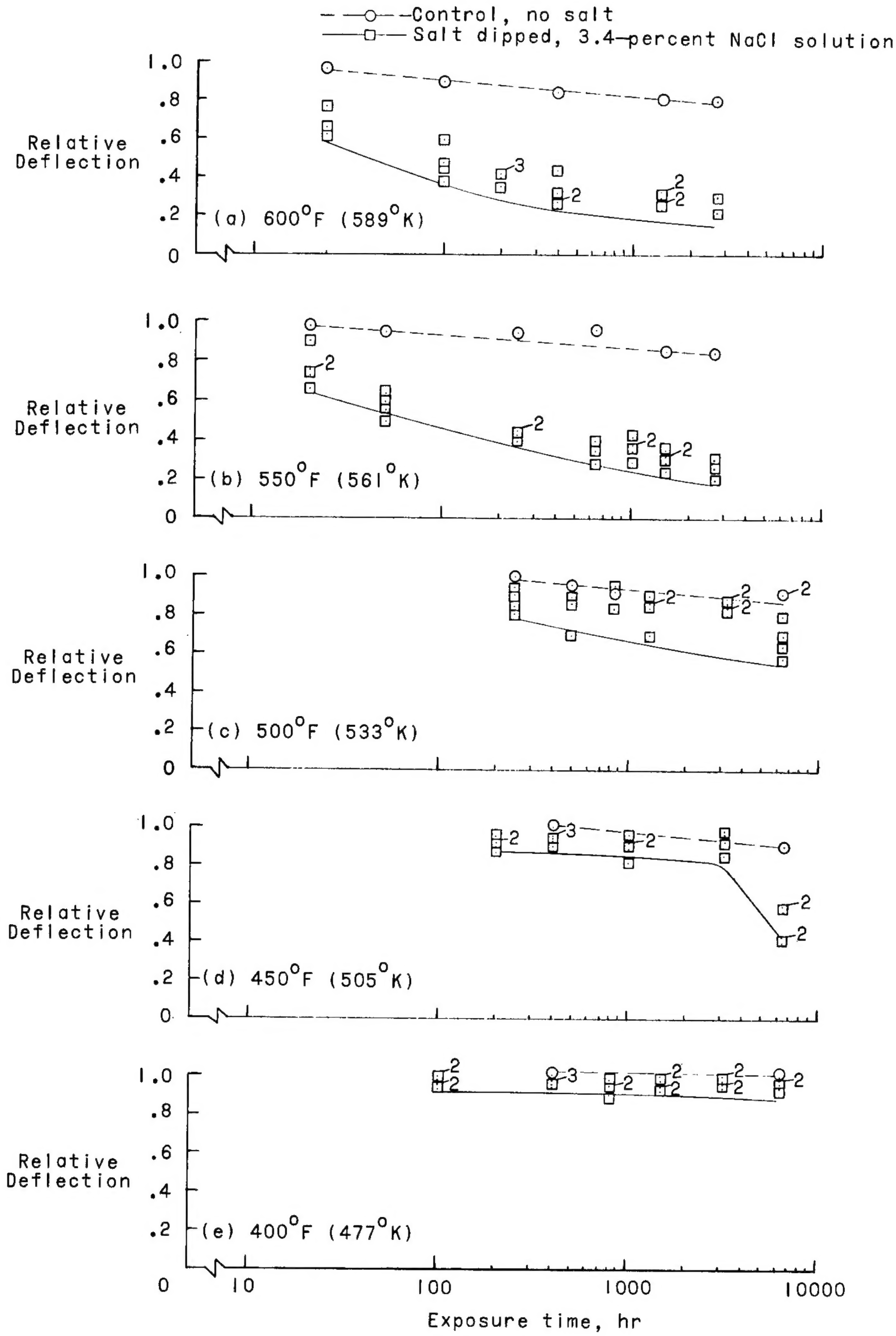


Figure 4.- Effect of various temperatures on salt stress corrosion of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for 0.25-inch-radius (0.6 cm) specimens.

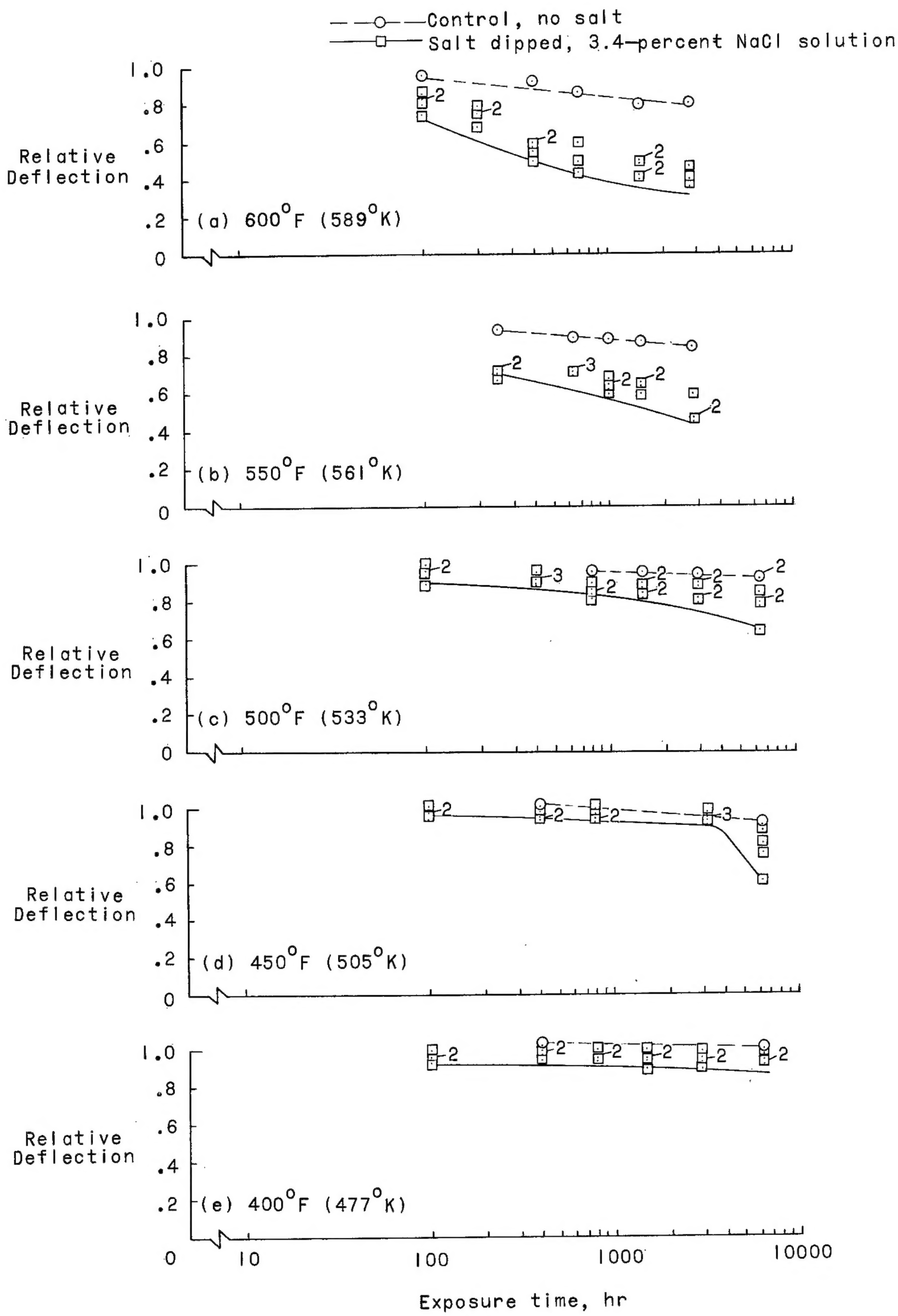


Figure 5.- Effect of various temperatures on salt stress corrosion of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for 0.5-inch-radius (1.3 cm) specimens.

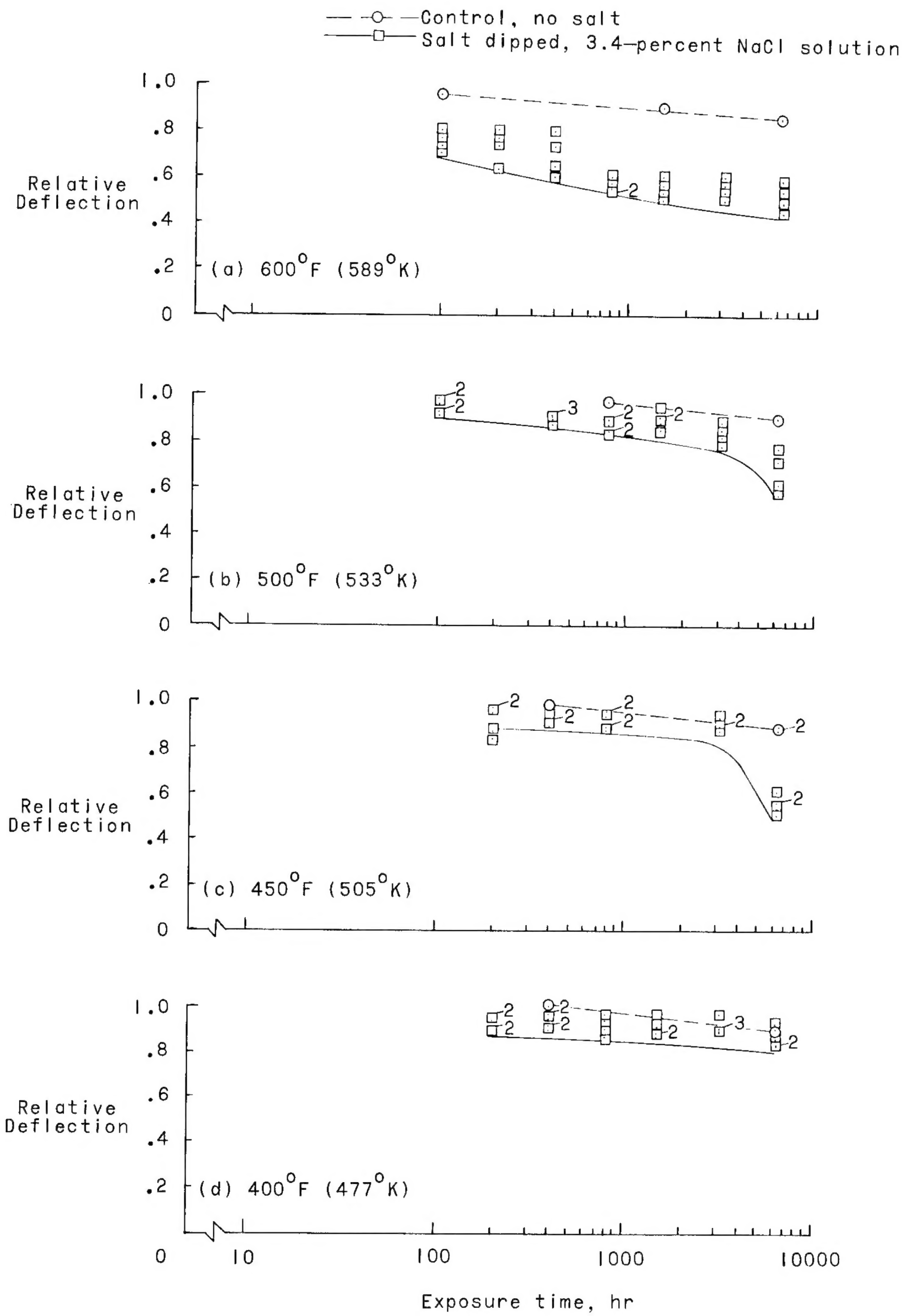


Figure 6.- Effect of various temperatures on salt stress corrosion of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for 1-inch-radius (2.5 cm) specimens.

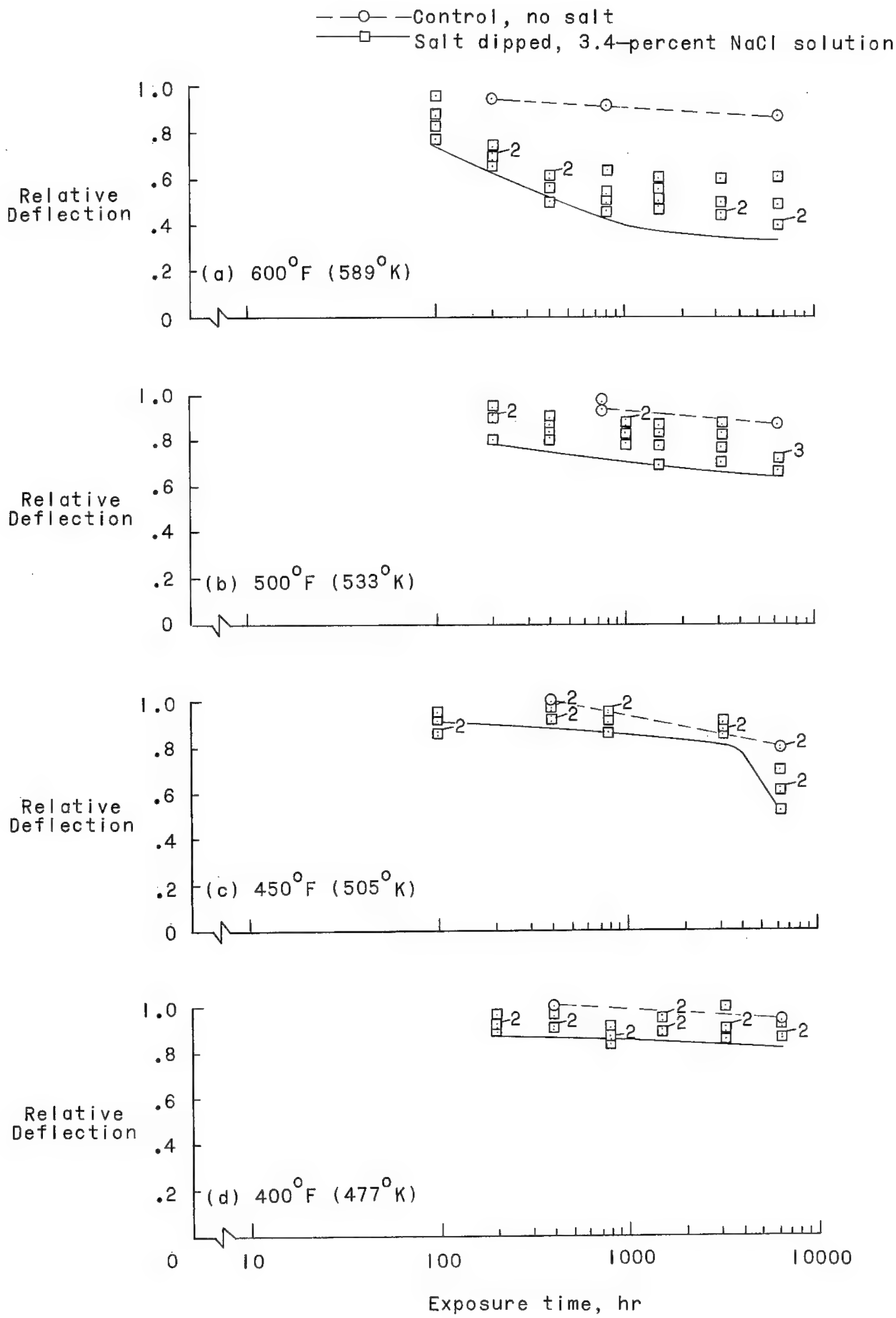


Figure 7.- Effect of various temperatures on salt stress corrosion of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for 1.5-inch-radius (3.8 cm) specimens.

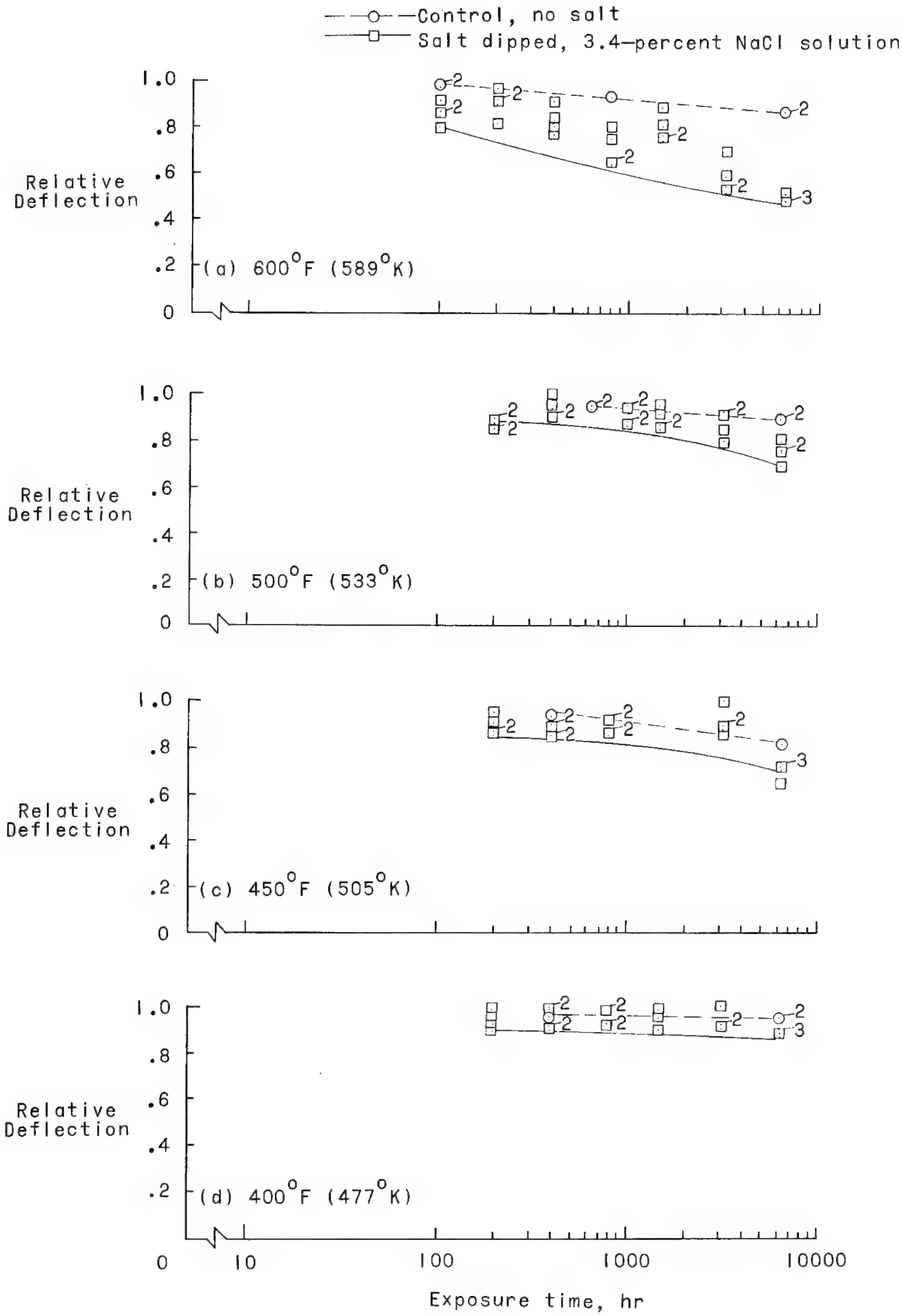


Figure 8.- Effect of various temperatures on salt stress corrosion of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for 2.5-inch-radius (6.4 cm) specimens.

The small reduction in deflection of the control specimens after exposure (figs. 4 to 8) appears to be due to a slight aging effect on the cold-worked duplex-annealed titanium. Metallurgical examination of control specimens after exposure showed no evidence of cracking.

The combinations of stress and temperature for which salt-stress-corrosion cracking begins are shown in figure 9. The curves in figure 9 represent only salt-stress-corrosion damage because they were obtained by determining the ratio of the relative deflection of the salt-dipped specimens to the relative deflection of the control specimens in figures 4 to 8. The results indicate that cracking begins at a temperature slightly above 400° F (477° K) for the 3200-hour as well as for the 6400-hour exposure. At 500° F (533° K) and 450° F (505° K) considerably more salt stress corrosion resulted after 6400 hours exposure than after 3200 hours exposure. Figure 9 shows that the severity of salt stress corrosion increases with an increase in temperature. The 0.25-inch-radius (0.6 cm) specimens are already severely cracked after 3200 hours exposure at 600° F (589° K); therefore, there was no need to continue exposure of these specimens at 600° F (589° K) beyond that time. The 2.5-inch-radius (6.4 cm) specimens show only a slight amount of salt stress corrosion at 600° F (589° K). Very little difference is observed between the data for the 3200- and 6400-hour exposures at 600° F (589° K), but a marked increase in salt stress corrosion is observed at 450° F (505° K) after the longer exposure. Results shown in figure 9 indicate that salt stress corrosion may be expected for exposure times of 3200 hours or more at temperatures slightly above 400° F (477° K) for the range of bend radii investigated.

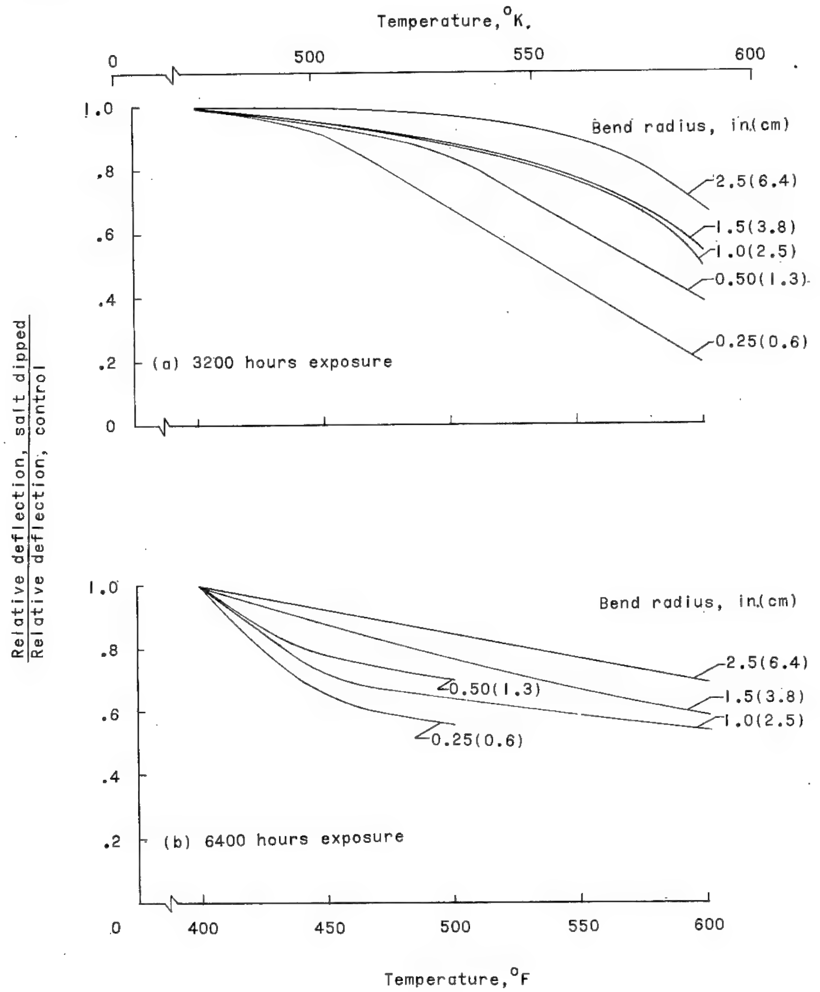


Figure 9.- Temperatures for which cracking begins for residually stressed duplex-annealed Ti-8Al-1Mo-1V after 3200 and 6400 hours exposure.

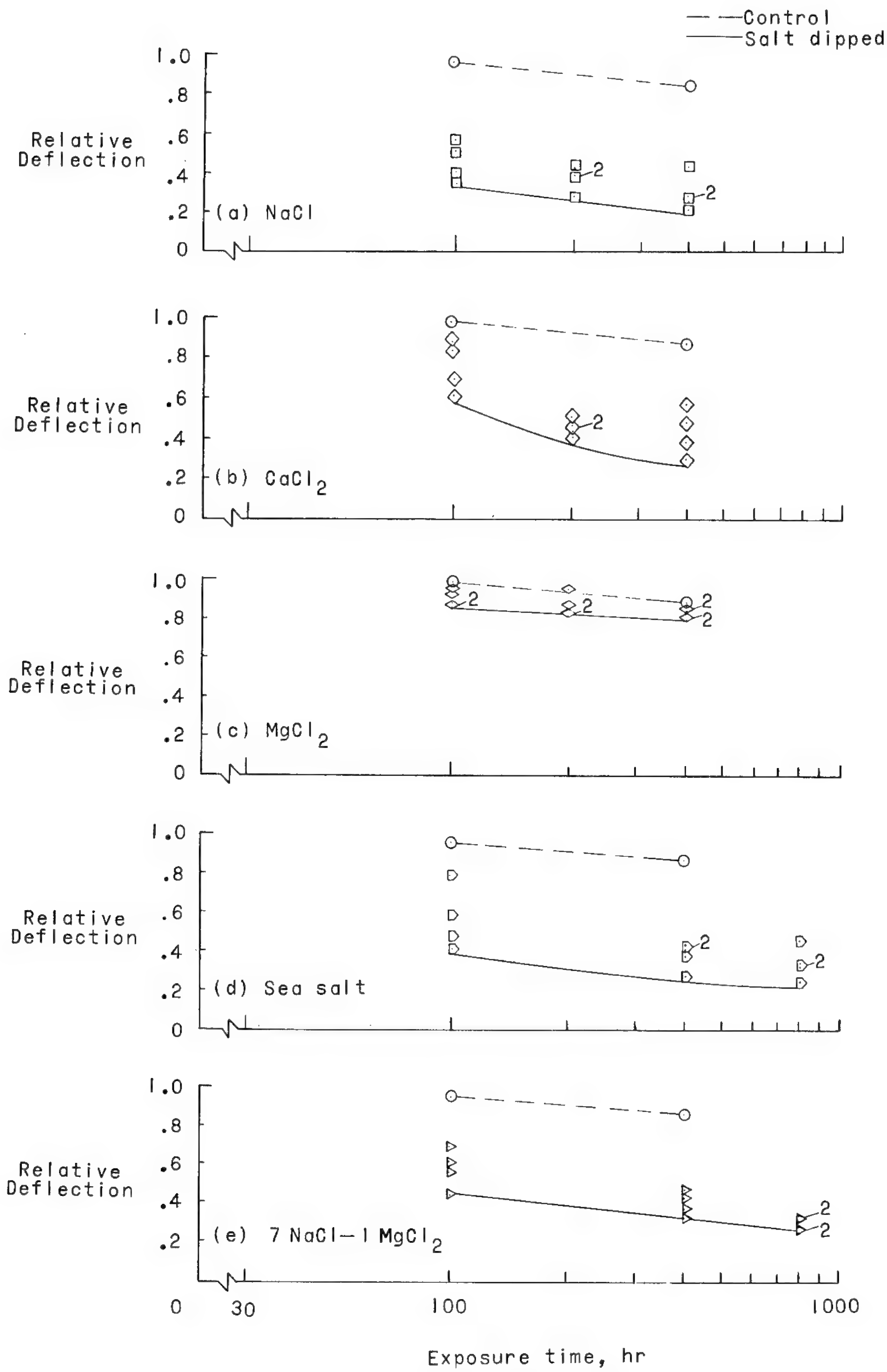


Figure 10.- Effect of various chlorides on stress-corrosion cracking of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for the 0.25-inch-radius (0.6 cm) specimens at 600° F (589° K).

The magnitudes of the relative deflection for the specimens with radii of 1, 1.5, and 2.5 inches (2.5, 3.8, and 6.4 cm) are about the same for both exposure times at 600° F (589° K). Therefore, the growth rate of stress-corrosion cracks apparently decreases after exposure times of approximately 3000 to 4000 hours for specimens of this type. A possible explanation for this phenomenon is that the corrosion cracks propagate into a lower residual tensile-stress area.

Effect of Various Chlorides

The effects of NaCl, $CaCl_2$, $MgCl_2$, sea salt, and simulated sea salt (seven parts NaCl to one part $MgCl_2$) on the salt stress corrosion of Ti-8Al-1Mo-1V alloy sheet (duplex annealed) at 600° F (589° K) are shown in figure 10 for the 0.25-inch-radius (0.6 cm) specimen. The curves shown are drawn through the approximate lower limit of the data. The data for the control specimens show very little change in relative deflection for the exposure times involved in these tests. Figure 11 shows the relative effects of the various chlorides on salt stress corrosion. The curves were obtained from the lower-limit data of figure 10. The most corrosive of all the salts investigated was NaCl, whereas the least corrosive was $MgCl_2$.

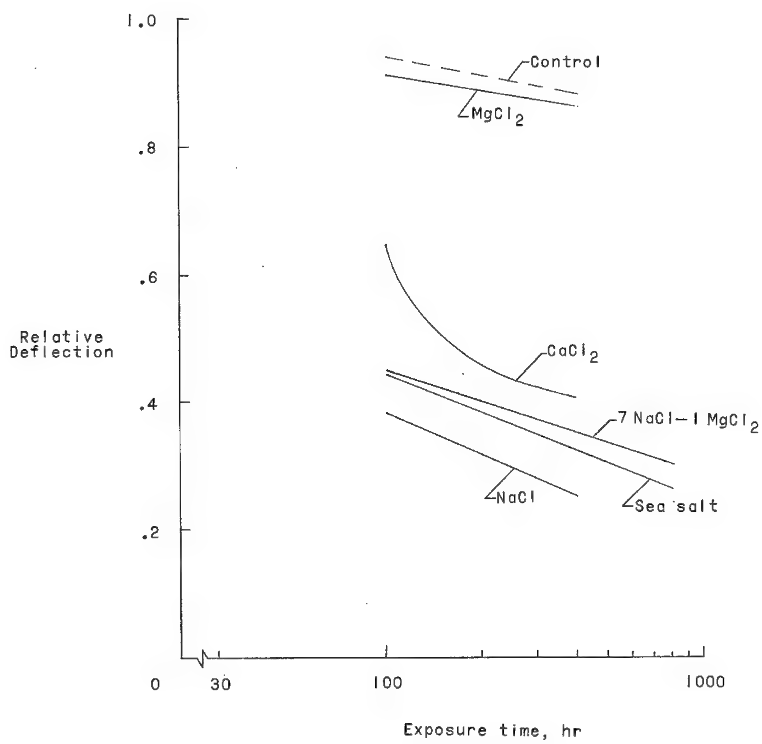
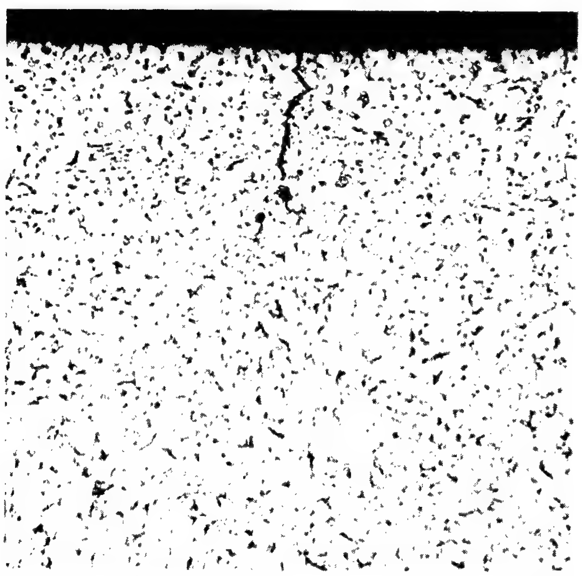


Figure 11.- Relative effect of various chlorides on stress-corrosion cracking of duplex-annealed Ti-8Al-1Mo-1V alloy sheet for the 0.25-inch-radius (0.6 cm) specimens at 600° F (589° K).

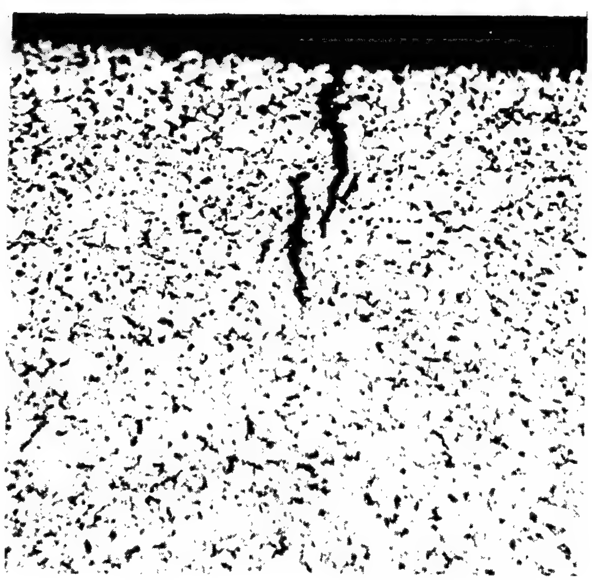
Metallurgical Examination

Photomicrographs of stress-corrosion cracks in the tensile region of residually stressed specimens after various exposure times for different radii and stress levels are shown in figure 12. These cracks and all those examined microscopically are generally intergranular. Figure 12(a) shows a fine salt-stress-corrosion crack which developed in a 0.25-inch-radius (0.6 cm) specimen after only 20 hours exposure at 600° F (589° K). Figure 12(b) illustrates crack discontinuity in a 0.5-inch-radius (1.3 cm) specimen which was exposed for 6400 hours at 450° F (505° K). The cracks shown were measured after testing the specimen by the reverse-bend procedure, but several specimens examined before testing had cracks just as deep as those measured after testing.

The results of the crack-depth measurements for the 0.25-inch-radius (0.6 cm) specimens after various times of exposure at 600° F (589° K) are shown in figure 13. The width of the band at any given exposure time shows that considerable variation exists between the shortest and deepest cracks. Difficulty in measurement makes correlation between maximum crack penetration and bend deflection difficult.



(a) 0.25-inch-radius (0.6 cm) specimen. 20 hours at 600° F (589° K); X 500.



(b) 0.5-inch-radius (1.3 cm) specimen. 6400 hours at 450° F (505° K); X 500.

Figure 12.- Typical salt-stress-corrosion cracking of duplex-annealed Ti-8Al-1Mo-1V alloy sheet. L-65-9038

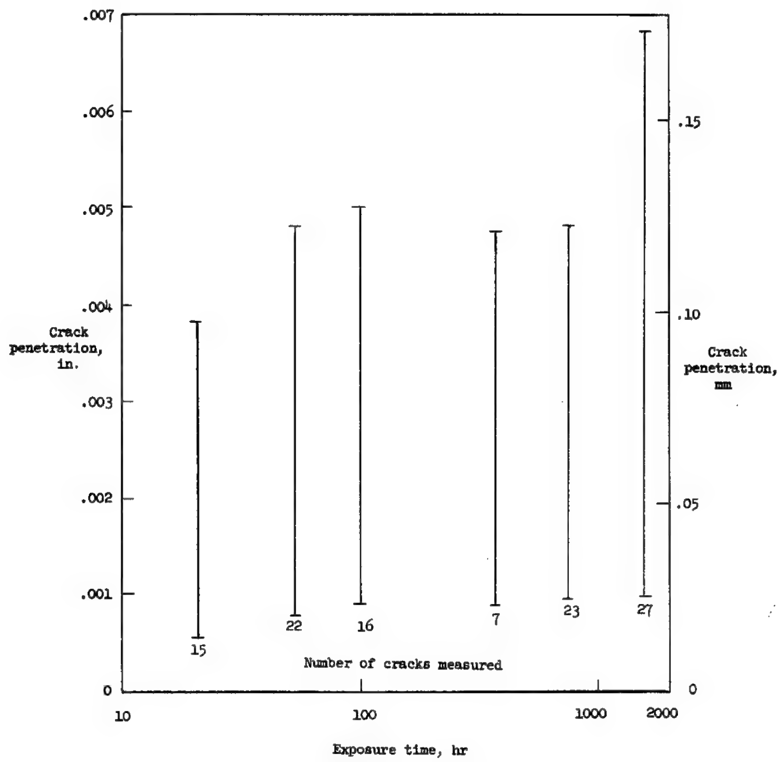
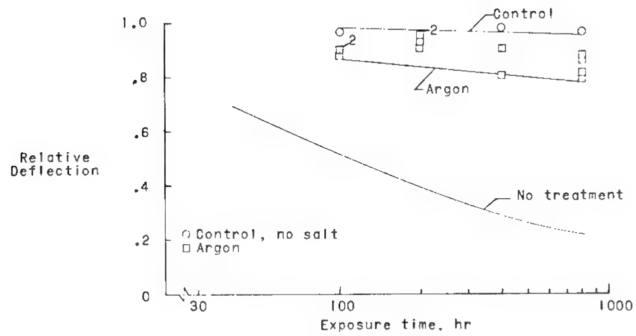


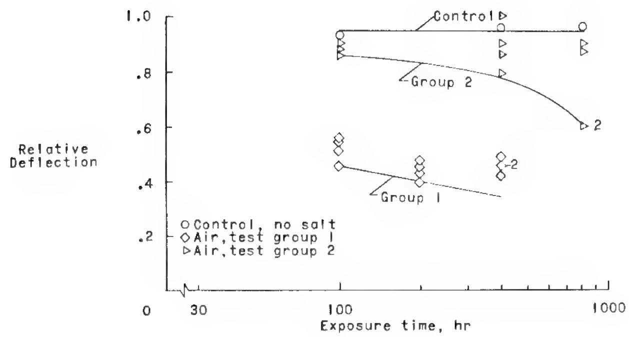
Figure 13.- Depth of crack penetration into residually stressed duplex-annealed Ti-8Al-1Mo-1V alloy sheet after various exposure times at 600° F (589° K) for the 0.25-inch-radius (0.6 cm) specimens.

Alleviation of Residual Stresses

Figure 14 shows the results for specimens stress relieved in air and argon. Stress relieving in argon (fig. 14(a)) seems to reduce residual tensile stresses enough to inhibit salt stress corrosion. The data shown in figure 14(a) are typical of the results for three groups of specimens stress relieved in argon. The curves shown are drawn through the lower limit of the test data. The results for the specimens stress relieved in air were not consistent; therefore, several groups of specimens stress relieved in air were investigated. Of all the groups stress relieved in air, only one group showed the annealing to be effective in inhibiting salt stress corrosion. Group 1 (fig. 14(b)) is typical of several groups run in air that did not inhibit corrosion to any significant extent. These results are comparable to those reported in reference 5. The fact that the stress-relief treatment in argon was effective but the treatment in air was not consistently effective suggests the possibility that contamination due to oxygen or other surface diffusion or contamination may be responsible for the susceptibility to salt stress corrosion. An explanation for the difference between the results of test groups 1 and 2 (fig. 14(b)) is not evident.



(a) Stress relieved in argon.



(b) Stress relieved in air.

Figure 14.- Effects of stress-relief treatments in argon and air on salt stress corrosion of 0.25-inch-radius specimen of duplex-annealed Ti-8Al-1Mo-1V exposed at 600° F (589° K).

CONCLUDING REMARKS

The salt stress corrosion of residually stressed Ti-8Al-1Mo-1V alloy sheet (duplex annealed) was investigated for exposure times up to 6400 hours at temperatures from 400° to 600° F (477° to 589° K). Salt stress corrosion was detected after 20 hours exposure at 600° F (589° K) for the 0.25-inch-radius (0.6 cm) specimens. Salt stress corrosion can be expected for exposure times of 3200 hours or more at temperatures slightly above 400° F (477° K) for the bend radii investigated. $NaCl$, $CaCl_2$, sea salt, and simulated sea salt (seven parts $NaCl$ to one part $MgCl_2$) proved to be very corrosive, whereas $MgCl_2$ caused little or no corrosion. Stress relieving was effective in alleviating stress corrosion when performed in an argon environment at 1450° F (1061° K) for one hour, but the results were nonconclusive when the specimens were stress relieved in air at the same temperature.

Langley Research Center,
National Aeronautics and Space Administration,
Langley Station, Hampton, Va., November 8, 1965.

APPENDIX A

CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

The International System of Units (SI) was adopted by the Eleventh General Conference on Weights and Measures held in Paris, October 1960, in Resolution No. 12 (ref. 6). Conversion factors required for units used herein are given in the following table:

Physical quantity	U.S. Customary Unit	Conversion factor (*)	SI unit
Force	pounds (lb)	4.448222	newton (N)
Length	inches (in.)	2.54	centimeters (cm)
Stress	kips per square inch (ksi)	6.896	meganewtons per square meter (MN/m^2)
Temperature	(^{o}F + 459.67)	5/9	degrees Kelvin (^{o}K)

*Multiply value given in U.S. Customary Unit by conversion factor to obtain equivalent value in SI unit.

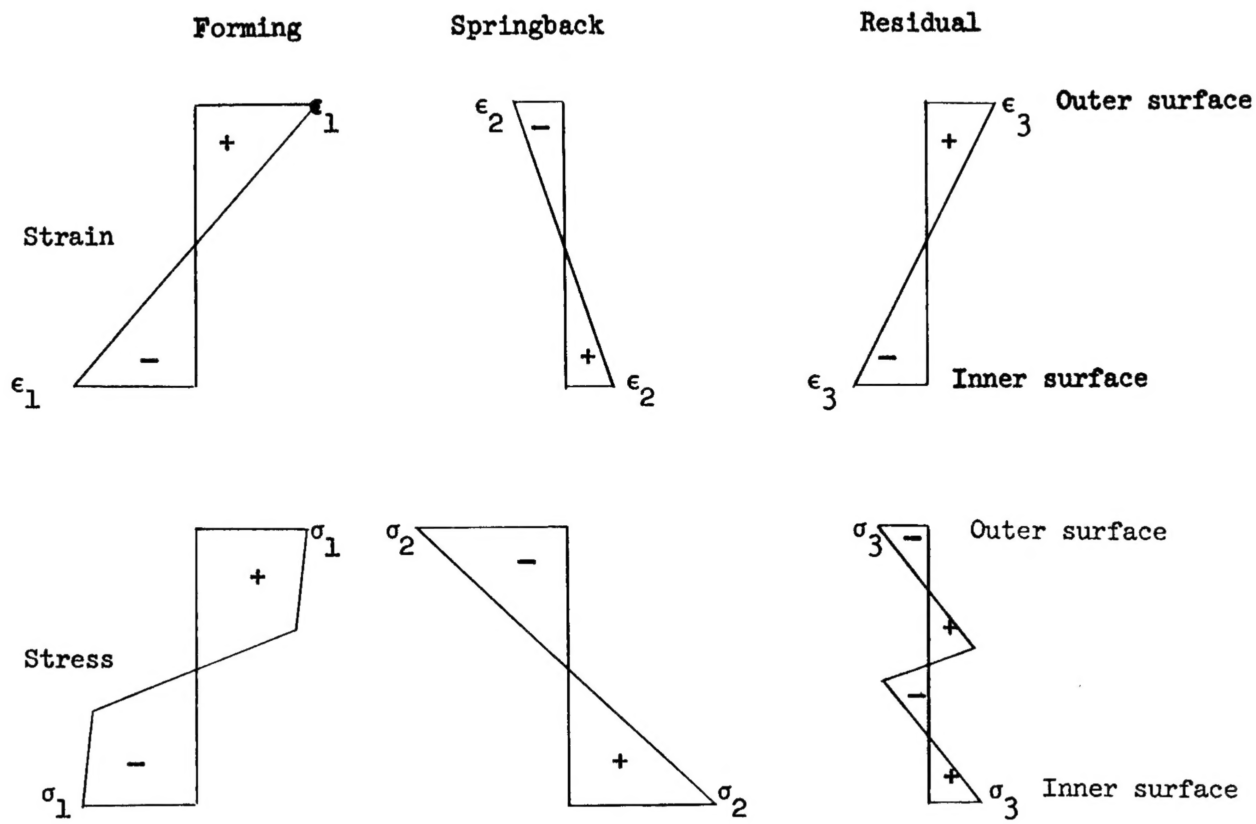
Prefixes to indicate multiples of units are as follows:

Prefix	Multiple
mega (M)	10^6
centi (c)	10^{-2}
milli (m)	10^{-3}

APPENDIX B

CALCULATION OF RESIDUAL TENSILE STRESSES

The calculation of residual tensile stresses was made by using the method described in reference 7. An outline of this method is as follows: The tensile stress-strain curve used in the calculations is given in reference 5. Tensile and compressive stress-strain relationships were assumed to be the same for duplex-annealed Ti-8Al-1Mo-1V sheet. Typical strain and stress distributions during and after the forming operation are shown in the following sketches:



The forming strain ϵ_1 in the outer fiber was obtained from the relationship

$$\epsilon_1 = \frac{t}{2\left(R + \frac{t}{2}\right)}$$

where

t sheet thickness

R bend radius

APPENDIX B

The forming stress σ_1 in the outer fiber was read from the stress-strain curve at the forming strain ϵ_1 in the outer fiber. The springback stress σ_2 in the outer fiber was obtained from the relationship

$$\sigma_2 = \frac{M\left(\frac{t}{2}\right)}{I}$$

where

M springback moment (equal and opposite to the forming moment)

I moment of inertia of section

and where the forming moment M was obtained from the relationship

$$M = \int_{-t/2}^{t/2} \sigma y \, dy$$

where

σ forming stress at any point

y distance from neutral axis

The springback strain ϵ_2 was obtained from the relationship

$$\epsilon_2 = \frac{\sigma_2}{E}$$

where

E Young's modulus

The residual stress σ_3 and strain ϵ_3 were obtained from the relationships

$$\sigma_3 = \sigma_1 + \sigma_2$$

$$\epsilon_3 = \epsilon_1 + \epsilon_2$$

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